Final Technical Report: On-line Oxidation of Volatile Organic Compounds Generated by Sawmill Wood Kilns

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Executive Summary

The primary purpose of this project was to evaluate the effectiveness of the use of hydrogen peroxide in combination with ultra-violet (UV) light for the reduction in the amount of volatile organic compounds (VOC's) released into the environment during wood drying in sawmill kilns. As originally envisioned the scope of work included the optimization of reaction parameters i.e. pH, peroxide concentration, UV light intensity that would lead to successful destruction of VOC's. The second phase involved development of a prototypical reactor that could be taken to a sawmill kiln for demonstration of treatment of a slipstream. However, after some of our initial experiments, we were forced to limit ourselves to more modest testing and development. During the project period we designed and constructed a small-scale reactor that was connected to the off-gas from a small wood drying kiln located at the Forest Products Lab at Mississippi State University. We ran a number of tests where we varied the process parameters (peroxide concentration, pH and UV light intensity) with no reasonable amount of VOC destruction. We employed a number of ways to atomize the peroxide into the kiln off-gas including using Laskin nozzles to see if that would improve the VOC reduction, but it was to no avail. The next option we explored was to try and utilize Fenton's Reagent to see if that would be an effective tool in our treatment process. Initial measurements appeared to provide very good VOC reductions, but upon more detailed investigation it appeared that the oxygen being generated in the Fenton's reaction was artificially reducing the VOC concentration coming from the treatment chamber. The last treatment system that we tried to use was the treatment of the kiln off-gas with hydrogen peroxide injection in the vapor phase. We were not able to achieve any success with this treatment method either.

It appears from our tests that there doesn't seem to be an easy way of obtaining VOC reduction using hydrogen peroxide in the presence of UV light. Either the reaction times are too long, or it is very difficult to get full mixing between the hydrogen peroxide and the off-gas.

Background

About thirty thousand short tons of VOC's are released into the environment during kiln drying of softwood species, in particular southern yellow pine. The traditional methods for controlling VOC emission, such as regenerative thermal oxidizers (RTO), recuperative catalytic oxidizers (RCO), or bioreactors are either expensive or very difficult to apply due to the large variations in humidity and VOC flow that occur during the drying process. In particular, the humidity fluctuations reduce the effectiveness of oxidation-type control devices and are detrimental to the organisms in bioreactors, which are also affected by temperature variations and interruptions in VOC output.

The primary technique evaluated in our work is a form of chemical oxidation referred to as an "Advanced Oxidation Process" (AOP). In this technique, UV photolysis of hydrogen peroxide results in the formation of hydroxyl radicals that oxidize or break down the organic and inorganic pollutants contained in the kiln emissions. UV-enhanced hydrogen peroxide oxidation is a generally accepted treatment method for removing organic constituents as well as for killing or deactivating micro-organisms in water. It's applicability to the treatment of gas phase VOC's is more limited and can be utilized efficiently only in very specific instances.

Chemical oxidation techniques are commonly employed to remove organic pollutants as part of wastewater treatment processes[1-6]. Processes have been developed and demonstrated for oxidizing contaminants such as toluene[7], acetone[8], and aromatics and aliphatics (and numerous other RCRA listed species) in industrial process wastewater[5, 6, 9-11]. Likewise, ground water contaminated with explosives (run off drainage from military firing ranges) has been successfully remediated by chemical oxidation of organic constituents[12].

The operation of wood kilns results in the release of monoterpenes, $C_{10}H_{16}$, a commercial source of turpentine[13]. Monoterpenes consist of isoprene units, C_5H_8, and are classified as regular cyclohexanoids[14]. Other volatile organics, α-pinene, β-pinene, limonene, β-myrcene, camphene and estragole have also been shown to be steam-volatile chemical compounds from pines[15]. These organic compounds are the pollutants of interest. All appear susceptible to chemical oxidation.

The methodology for pollutant destruction is a form of chemical oxidation referred to as "Advanced Oxidation Processes (AOP)". AOPs are described as chemical oxidation processes that utilize the formation of the hydroxyl radical ($OH^{\cdot}$) to oxidize or break down the organic or inorganic contaminants. The most commonly used AOPs involve ultra-violet (UV) light photolysis of hydrogen peroxide (H_2O_2), UV photolysis of ozone (O_3), or peroxone (a chemical system employing a combination of ozone and hydrogen peroxide)[8].

Of these three AOPs, the UV photolysis of H_2O_2 is the most attractive for several reasons.

1) Photodecomposition of H_2O_2 is a highly proficient mechanism for the formation of the hydroxyl radical[16].

$$H_2O_2 + h\nu \rightarrow 2OH^{\cdot}$$

2) Hydrogen peroxide is an effective oxidizing agent when coupled with UV irradiation and is relatively inexpensive.

3) Hydrogen peroxide is also stable and easily mixed with water.

Ozone is relatively expensive to produce at low levels and is an unstable gas that requires on-site production and transferal into an aqueous solution[9]. With respect to commercialization, hydrogen peroxide processes are generally less sensitive than ozone during process scale-up operation[9].

Hydrogen peroxide splits into hydroxyl radicals when irradiated with UV light. These hydroxyl radicals are highly reactive (see Table 1) and attack organic substrates by abstracting hydrogen atoms or by adding to the double bonds of unsaturated molecules[8].

$$M_{organic} + OH^{\cdot} \rightarrow \text{Products}$$

Ideally, the resulting products will be water, carbon dioxide, and low molecular weight aliphatic acids[9]. Intermediate products may also form; it has been shown that intermediates can often be eliminated by adding surplus peroxide and increasing the reaction time [9, 17].

Table 1: Comparison of common oxidizing agents used in water treatment[8].

Oxidizing Species	Oxidation Potential (Volts)
Hydroxyl Radical	2.8
Ozone	2.1
Hydrogen Peroxide	1.8
Potassium Permanganate	1.7
Hypochlorous acid	1.5
Chlorine Dioxide	1.5
Chlorine	1.4
Oxygen	1.2

In addition to reacting with the pollutant or contaminant of interest, hydroxyl radicals may react with other constituents of the matrix, namely inorganics and reduced cations[8]. These unwanted side or "scavenging" reactions can significantly reduce the ability of the hydroxyl radical to oxidize the desired contaminant[8]. Carbonate, bicarbonate, phosphate (inorganic mineral scavengers) and iron (reduced cation scavenger) are some of the more common competitors for oxidation by the hydroxyl radical. The inhibiting effect is directly proportional to scavenger concentration[8].

$$OH^{\cdot} + HCO_3^{-} \rightarrow OH^{-} + HCO_3^{\cdot}$$
$$OH^{\cdot} + CO_3^{2}\text{-} \rightarrow OH^{-} + {\cdot}CO_3^{-}$$
$$OH^{\cdot} + PO_4^{3-} \rightarrow OH^{-} + {\cdot}PO_4^{2-}$$

The UV light source of choice for most UV-AOP (peroxide based) systems is the medium pressure mercury vapor lamp (MPUV). MPUVs emit significant radiation in the range of 200 to 250 nm, the region of strong absorption for H_2O_2[8].

A number of parameters affect the production of hydroxyl radicals by UV photolysis. These include pH, temperature, H_2O_2 concentration, and scavenger species[8]. Solution pH can affect the generation of hydroxyl radicals since H_2O_2 is a weak acid and slightly ionizes in water via two steps.

$$H_2O_2 \leftrightarrow H^{+} + HO_2^{-} \text{ (hydrogen peroxide ion)}$$

$$HO_2^{-} \leftrightarrow H^{+} + O_2^{2-} \text{ (peroxide ion)}$$

The occurrence of this reaction is unwanted because it expends hydrogen peroxide without producing hydroxyl radicals[8].

Another critical variable in the generation of hydroxyl radical is the actual concentration of hydrogen peroxide. Ultimately, the formation of hydroxyl radicals is limited due to the fact that H_2O_2 in high concentrations can act as a scavenger itself[8, 16, 18].

$$H_2O_2 + OH^{\cdot} \rightarrow H_2O + HO_2^{\cdot}$$

In practice, therefore, the concentration of H_2O_2 should be optimized so that the rate of reaction between the pollutant and the hydroxyl radical is greater than the rate of reaction between the hydroxyl radical and the parent oxidizer, H_2O_2[8]. Due to the critical aforementioned variables, it is of the utmost importance to optimize the hydrogen peroxide concentration and pH while minimizing the effects of scavengers that may be present in the matrix. Otherwise, the cost efficiency of the process is needlessly reduced.

As a first step in the investigative process, we designed and constructed a reactor to study the treatment of the VOC's being emitted in the wood-drying process from a small laboratory sized kiln. The reactor served a dual purpose in quenching the off-gas to reduce the moisture content as well as to provide a flexible platform from which to study the interaction of UV light from low-pressure and medium pressure lamps on a hydrogen peroxide stream to treat the off-gas. At first, the peroxide was injected using a common nozzle to form tiny droplets and increase the contact area for the reaction to take place. A variety of hydrogen peroxide concentrations were tested. We then utilized an ultra-sonic nozzle and a Laskin nozzle (micron size to sub-micron size aerosol generator) to treat the VOC's, using secondary treatment chambers with UV lamps. All of these efforts involved the (peroxide) liquid phase treatment of the VOC's. We were unable to get any significant reduction in the VOC treatment process using any of the techniques.

The second technique that we utilized was employing Fenton's Reagent for the oxidation of the VOC's. Fenton's Reagent is the combination of a ferrous (Fe^{2+}) or ferric (Fe^{3+}) salt with hydrogen peroxide. Iron from the ferrous or ferric salt is the catalyst in the reaction which produces the highly desirable hydroxyl free radicals ($HO^{\bullet}$) or hydroperoxyl free radicals ($HO_2^{\bullet}$) used in advanced oxidative processes (AOPs).

A number of tests were carried out on the large reaction chamber with what appeared to be very encouraging results. We then utilized smaller reaction chambers more amenable to the control of the reaction but with no success. As it turned out, the vast quantities of oxygen generated during Fenton's reaction was preventing the off-gas from entering the big reaction chamber and thus give us an artificially low measurement of VOC at the exit of the large treatment chamber.

The third and last technique that we utilized was in the (peroxide) vapor phase treatment of the VOC's. Hydrogen peroxide flowing in a stainless steel tube was vaporized in an oil bath and injected into the quenched off-gas stream (downstream of the main treatment vesesel) into a secondary treatment chamber. We employed varying concentrations of hydrogen peroxide with both the low pressure and medium pressure UV lamps, but with no success. We then tried to employ this same treatment process immediately at the exhaust of the kiln (pre-quenching) but did not obtain any success in reducing the VOC's.

Most of this experimental work was conducted at the Forest Products Research Center at MSU, led by Dr. Phil Steele and Dr. Leonard Ingram who contributed their wood-drying kiln, off-gas testing equipment and other resources to the effort. The team has many years of research experience in the wood/forest products industry. The DIAL/MSU team was led by Dr. J.P. Singh and R. Arun Kumar. Dr. J.P. Singh has a wide variety of experience in the utilization of optical techniques for emissions measurement. Mr. R. Arun Kumar has been the Chief Test engineer at DIAL and his expertise is in heat transfer, off-gas treatment methods, instrumentation and testing. Dr. John Walton of U.S. peroxide, our industrial partner in this project, has many years of experience in the peroxide industry and is an expert on the utilization and properties of hydrogen peroxide. In addition, the analytical laboratory at DIAL was involved in the analysis of reaction conditions and in the preparation of solutions that were used on the project.

Work Performed

The primary reaction vessel designed for the VOC treatment process was a combination of a mini packed column to knock out some particulates followed by the VOC treatment section. A schematic of the cell is shown in Figure 1. VOC laden air entered the bottom of the lower chamber from the left side. It flowed up through a layer of marbles into the upper treatment section. The marbles provided the dual function of distributing the air through the cross-section, as well as providing for an efficient liquid/gas interface with the recirculating peroxide solution to knockout some of the particulates. The gas then entered the treatment section which was a chamber 24" tall and 18" in diameter. A quartz tube ran across the middle of the chamber which housed the UV lamp that aided in the treatment of the VOC's. The treated gas then exited the top of the chamber. Gas sampling probes were located at the inlet and outlet of the test chamber in order to evaluate the VOC removal efficiency. The total height of test cell was 50". Peroxide solution from the reactor was drawn from the bottom of the lower section by a gear pump. On the inlet side of the pump were provisions for caustic/acid injection by a pH controller, as well as a recirculation and fill valve. The gear pump was a Teel Model IV454 with stainless steel housing and Viton gears with graphite bushings. It was driven by a DC 1/3 HP variable speed motor with a 5:1 gear reduction in between so as to keep recirculation pressures within 100 psig. We kept the downstream pressures between 35 and 80 psig which corresponded to recirculation flow rates of 0.25 to 0.36 gpm respectively. The outlet of the pump was connected to a domestic 5 micron water filter to knock out any recirculating particulates that would clog the spray nozzle. The clean solution then passed through a valve combination which permitted liquid sampling (to monitor peroxide concentration) during a test and included a bypass outlet allowing some of the clean solution to pass through a cooling section to provide temperature control. The cooled liquid was then pumped back to the test chamber facilitating thorough mixing of the solution. The re-circulating fluid then passed through a plumbing tree that housed a pressure gauge and a pH sensor (to provide for pH control), as well as a type T (Copper-Constantan) thermocouple to measure the fluid temperature. The solution was then re-circulated into the top of the test section through an injection nozzle. As the drying cycle progressed, the water in the exhaust gas from the wood drying kiln was knocked out in the test cell due to a decrease in gas temperature (quenching). In order to accommodate the water being condensed, as well as the caustic being injected into the test cell, an over-flow tap at 9" (operating level) from the bottom of the treatment cell was provided.

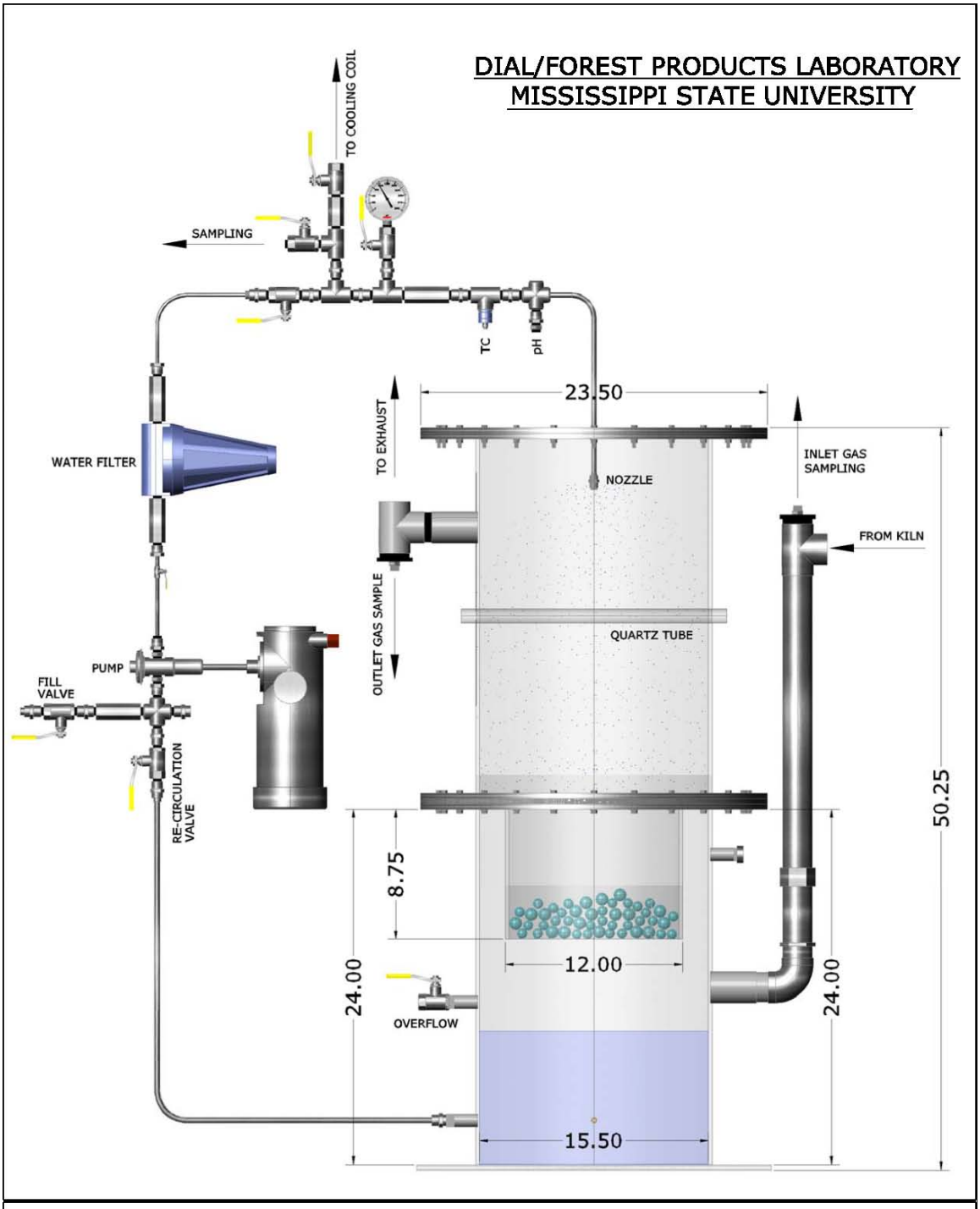

FIGURE 1
VOC TREATMENT FLOW PROCESS

Sample gas for measurements was extracted upstream and downstream of the reactor to measure VOC reduction efficiencies. Sample gas that was extracted passed through a set of impingers before analysis by dual J.U.M. VE7 total hydrocarbon analyzers. These analyzers employ Flame Ionization Detectors (FID) and provide good accuracy, linearity and different spans to suit measurement conditions. A number of other parameters were measured or logged during the course of every test, including the kiln gas temperatures, hydrogen peroxide flow pressures and temperatures, etc.

The kiln was loaded with 15 pieces of (2 by 4) green lumber stacked 3 across by 5 high with spacers between the rows. The purge air flow to carry the moisture out from the drying wood was then typically set to 1 cfm. The kiln was then turned on to ramp up to an operating temperature of 235 degrees F over a period of 2 hours and then remained constant for the remainder of the test. As the kiln temperature rose, there was a corresponding increase in the VOC concentration exhausting from the kiln. Maximum total hydrocarbons measured depended on the initial condition of the wood, but concentrations ranged anywhere between 1000 ppm to a little over 4000 ppm.

Figure 2 from Test 6 shows typical results from measurements made during a test.

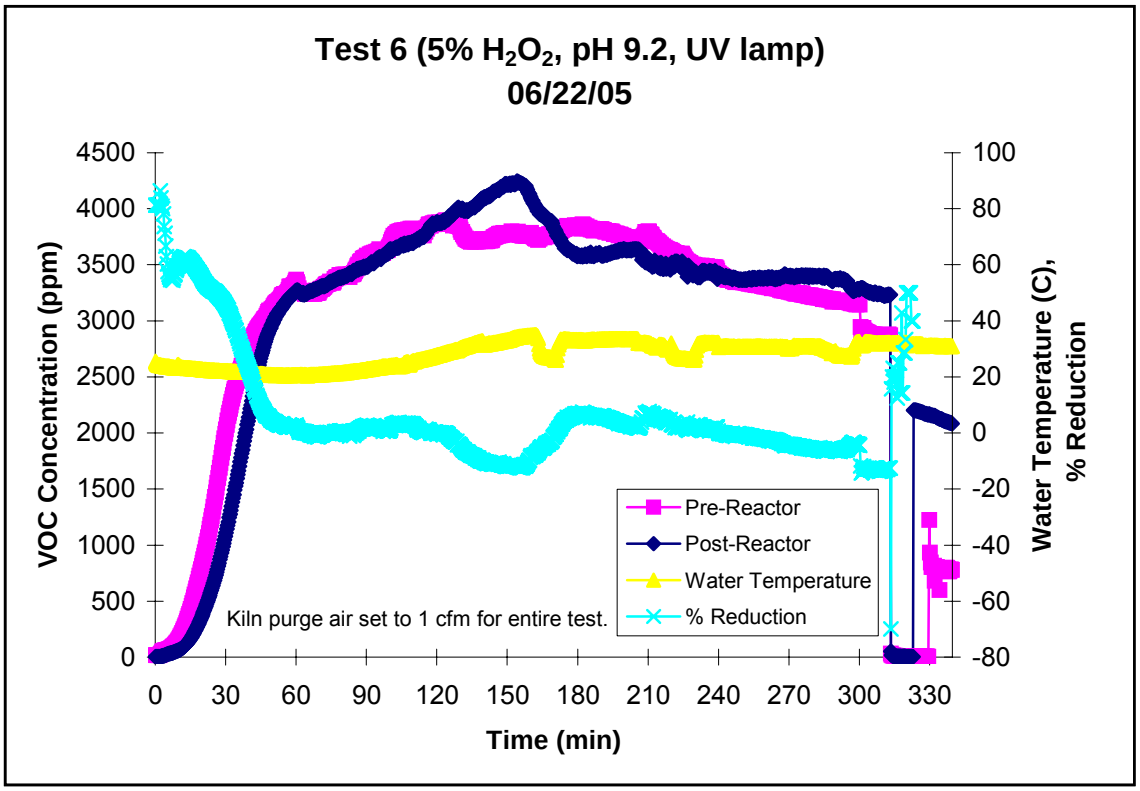

Figure 2. Scrubber operating parameters during Test 6

The range of hydrogen peroxide concentrations that were used for treatment in the first phase of the tests were between 0% and 7% with pH ranges between 3 and 10. Unfortunately, we were unable to achieve any significant reduction in the VOC's.

In order to increase the volumetric energy density of the UV lamp, a secondary treatment chamber was constructed as shown in Figure 3. This secondary chamber was installed downstream of the primary

chamber. It consisted of a 3 in. diameter, 16 in. long pipe with a 1 inch diameter quartz tube running down the middle of it to house the UV lamp. Two ports for injection of hydrogen peroxide liquid/vapor were provided.

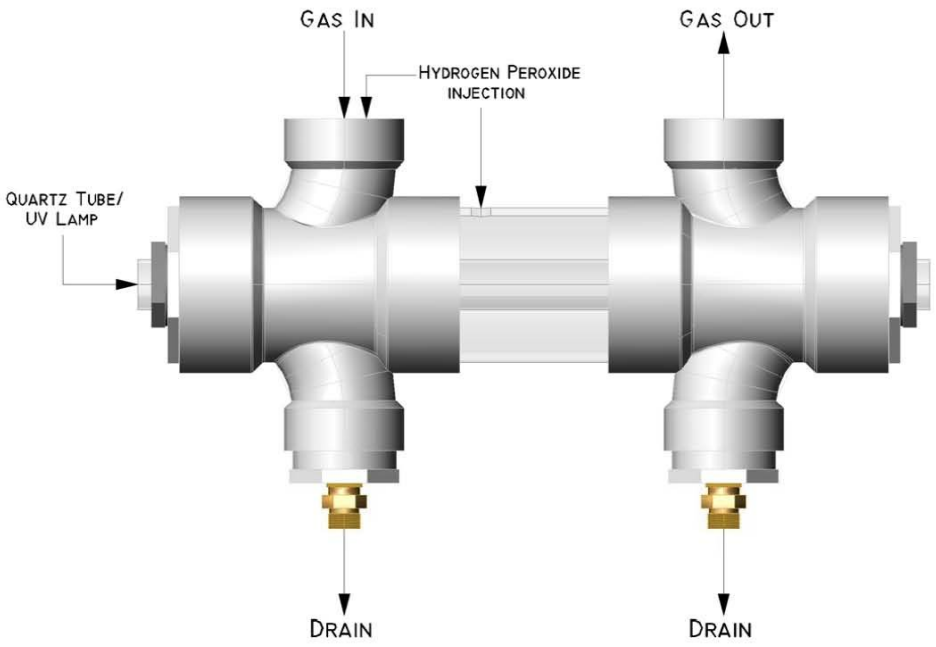

Fig 3: Schematic of secondary treatment chamber

The primary aim of this secondary chamber was to channel all the off-gas flow through a smaller volume closer to the UV lamp. This secondary treatment chamber had a treatment volume of about 100 cubic inches as compared to the primary vessel which had a 5000 cubic inch treatment volume. In this secondary chamber we utilized both an ultrasonic nozzle as well as a Laskin nozzle to inject the hydrogen peroxide into the flow stream. The Laskin nozzle required air injection for atomization which became part of the gas stream, thus it lowered the VOC concentration purely by dilution. Figure 4 shows some of the measurements made while testing this secondary chamber. There is no reduction in the VOC concentrations except during the time when the Laskin nozzle is utilized to inject the peroxide. This reduction appears to be the result of dilution air provided to the stream.

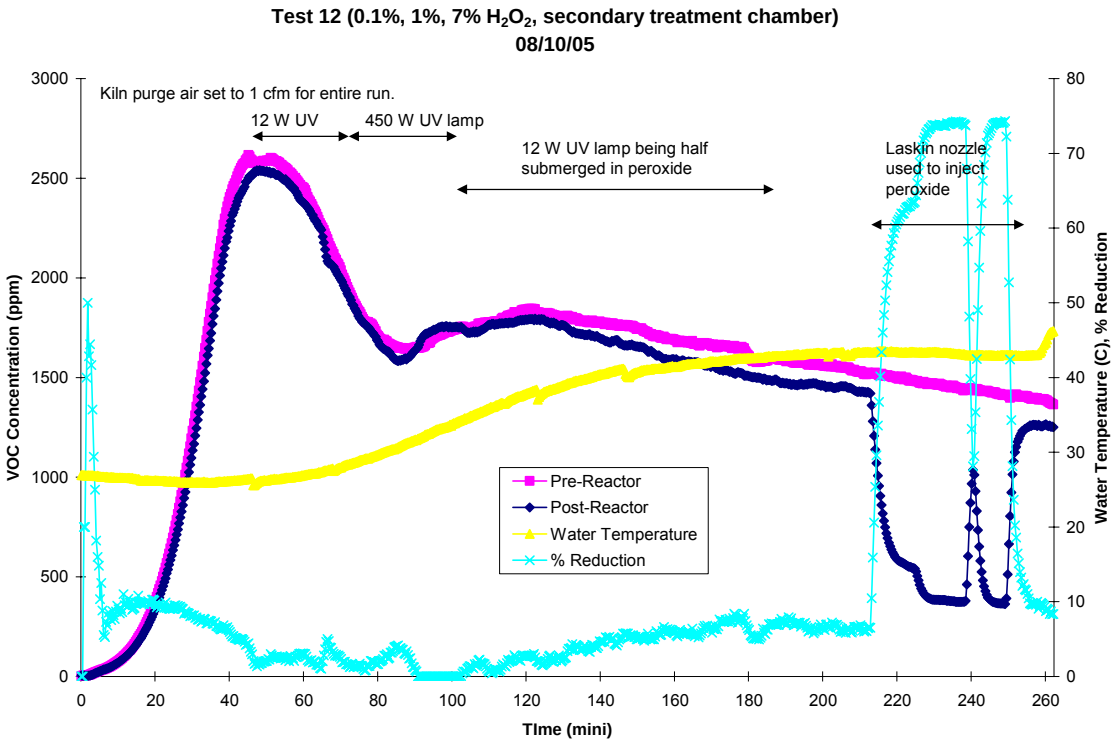

Figure 4. Scrubber operating parameters during Test 12

Having explored without success the liquid/aerosol phase-hydrogen peroxide treatment of the VOC's with the UV lamp, we also attempted to utilize Fenton's Reagent for the same purpose using the primary reaction vessel and subsequently some smaller secondary chambers.

Traditionally this reagent has been used in a number of waste-water treatment processes. The combination of a ferrous (Fe^{2+}) or ferric (Fe^{3+}) salt with hydrogen peroxide is commonly referred to as Fenton's Reagent[19]. Iron from the salt (typically either ferric sulfate or ferrous sulfate) is the catalyst in the reaction which produces the highly desirable hydroxyl free radicals ($HO^{\bullet}$) or hydroperoxyl free radicals ($HO_2^{\bullet}$) used in advanced oxidative processes (AOPs)[19].

(1) $Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + HO^- + HO^{\bullet}$

(2) $Fe^{3+} + H_2O_2 \rightarrow Fe^{2+} + HO_2^{\bullet} + H^+$

Fenton's Reagent is highly unstable and a number of additional reactions (see below) also occur in which other oxidants such as oxygen and hydrogen peroxide itself are produced. However, the hydroxyl radical has the highest oxidation potential, and is, therefore, the most sought after reaction product when dealing with AOPs[19].

(3) $Fe^{2+} + HO^{\bullet} \rightarrow Fe^{3+} + HO^-$

(4) $H_2O_2 + HO^{\bullet} \rightarrow H_2O + HO_2^{\bullet}$

(5) $Fe^{2+} + HO_2^{\bullet} \rightarrow Fe^{3+} + HO_2^- \leftrightarrow H_2O_2$

(6) $Fe^{3+} + HO_2^{\bullet} \rightarrow Fe^{2+} + H^+ + O_2$

A number of factors can influence the efficiency for production of hydroxyl radicals when dealing with Fenton's Reagent including the following: pH, temperature, iron concentration, peroxide concentration, and iron to peroxide ratio[20]. The most favorable pH for reaction efficiency occurs in the range of 3 to 6. At basic pH values, the iron forms the colloidal ferric species $Fe(OH)_3$ which decomposes the peroxide into oxygen and water[20]. This can lead to potentially dangerous and hazardous conditions if elevated levels of oxygen are produced.

Increasing temperature has an affect on Fenton's reactions by increasing the rate of reaction with the effect being more evident at lower temperatures (< 20°C). However, at temperatures above 40 to 50°C, the effect is less pronounced. Therefore, temperatures between 20 and 40°C are utilized most often for commercial uses.

Regarding iron and peroxide concentrations, the ideal is to find minimum concentrations of each that allow the catalytic reaction to proceed while still maintaining the upper limit for organic contaminant removal. Iron concentrations need to sustain a minimal threshold of usually 1 part Fe per 10 to 50 parts organic contaminant while preserving an iron to peroxide ratio on the order of 1 part iron per 5 to 25 parts peroxide (wt/wt)[20].

In some instances, additional iron may be required to exceed the chelating properties of the organic substrate in question to allow the excess iron to catalyze the peroxide for hydroxyl radical production. Likewise, supplementary peroxide may be needed to push the reaction past the point where unwanted organic intermediates form.

The cleaning agent used for test 9 was 5% peroxide (H_2O_2). The pH of the solution was initially set to 4. The UV lamps were not used for this test. Lumber was loaded into the kiln, and the kiln was heated to 235°F over a three hour period. The kiln purge air was set to 1 cfm for the entire test. Samples of the peroxide solution were taken throughout the test to monitor the concentration. A heat exchanger was used for cooling the circulating peroxide solution during this test.

200 minutes into the test, a 2% ferrous sulfate solution (Fenton's Reagent) was added to the peroxide solution. When the ferrous sulfate was added to the peroxide solution, the VOC concentration exiting the reaction vessel was greatly reduced for a period of 10 minutes. Titration measurements on the recirculating peroxide showed that the concentration decreased very quickly as well. This allowed the VOC concentration to begin increasing again. 255 minutes after the beginning of the run, the peroxide and ferrous sulfate solutions were replenished via the top of the chamber, and the VOC concentration was again reduced. The results from this test were very encouraging, so a series of tests were performed to determine the applicability of this technique using Fenton's reagent for the off-gas VOC reduction.

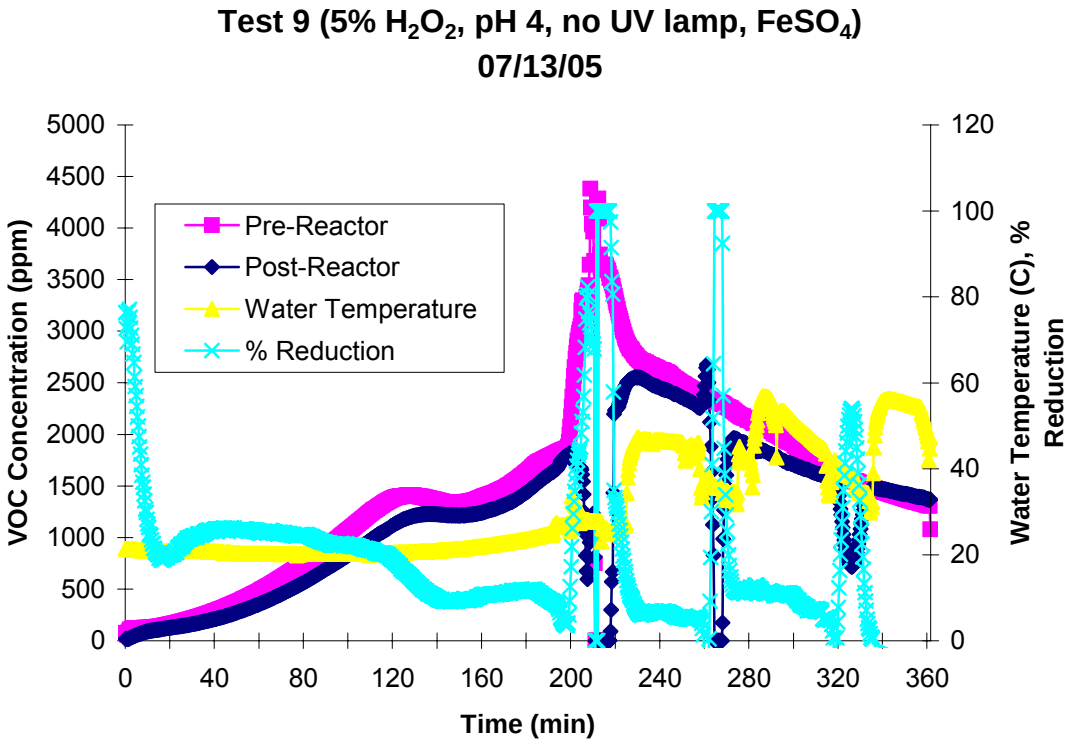

Figure 5. Scrubber operating parameters during Test 9

Laboratory-scale bench-top work was also conducted to verify if the effect of Fenton's was authentic or a result of kiln-gas dilution/ inhibition. In all the larger scale tests, the Fenton's reaction (exothermic) was accompanied by significant amounts of gas generation. The benchtop measurements showed that secondary gas generation/dilution by oxygen and hydrogen was the cause of the apparent VOC reduction. Substantial effort was also expended in this effort.

Having limited, if any, success with the liquid/aerosol treatment of the VOC stream with peroxide, we designed a system for generating hydrogen peroxide vapor for injection into a secondary chamber to see if that would improve the VOC treatment efficiency. Off-gas from the kiln was routed through the primary treatment vessel to reduce the moisture content as well as quench the gas. The off-gas was then passed through the secondary treatment chamber shown in Figure 3 for VOC reduction. Total hydrocarbon concentrations were measured at the inlet and the outlet of the secondary chamber to determine the VOC destruction efficiency. Hydrogen peroxide was pumped through a stainless steel tube immersed in an oil bath to vaporize the hydrogen peroxide. The hydrogen peroxide vapor was then injected into the secondary chamber to treat the VOC's in the presence of a UV lamp. Hydrogen peroxide concentrations of 3%, 5% and 10% were studied. Both the low pressure and medium pressure UV lamps were utilized to enhance the VOC oxidation. A 3% concentration of hydrogen peroxide vapor dropped the VOC concentration from 864 ppm to 769 ppm, an 11% decrease (with the low pressure UV lamp). The 5% peroxide injection had a similar decrease and turning off the UV lamp brought the downstream concentration up to 810 ppm, a 6.25% decrease. The 10% peroxide solution also had about a 10% reduction with the low pressure UV lamp in place. With the medium pressure lamp in place, VOC destruction efficiencies were in the 14% range with the upstream concentration of 849 ppm being reduced to 730 ppm.

In order to evaluate the treatment efficiency at elevated temperatures, the secondary treatment unit

(with UV lamp) was installed at the exit of the kiln. Gas from the secondary treatment unit was then passed through the primary chamber for quenching before being vented.

Total hydrocarbon concentrations were measured at the inlet and the outlet of the secondary chamber to determine the VOC destruction efficiency. Hydrogen peroxide concentrations of 10% and 20% were studied. The treatment was studied using both the low pressure and medium pressure UV lamps. VOC reductions were once again only marginal.

Accomplishments

During the course of this project, we have investigated the application of an advanced treatment process using hydrogen peroxide in the presence of a UV light to eliminate the volatile organic compounds produced in the off-gas stream from a wood-drying kiln. We designed, constructed and tested a main treatment vessel and several secondary vessels but had no success in reducing the volatiles present in the gas stream. We attempted to treat the gas stream with the hydrogen peroxide both in a liquid aerosol form as well as in the vapor form. We also attempted to use Fenton's Reagent for the VOC removal treatment but determined that our apparent good results were an artifact due to the generation of large volumes of oxygen and hydrogen.

Conclusions

Based on our testing we have come to the conclusion that utilizing UV light to help treat volatile organic compounds with hydrogen peroxide is going to be more complicated than originally envisioned. The residence times/UV energy intensity requirements may be too much for treatment of gas phase organics.

Recommendations

None.

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